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Indian Standard

METHODS OF TEST FOR PULP

PART XV DETERMINATION OF MANGANESE

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***February* 1976**

Indian Standard

METHODS OF TEST FOR PULP

PART XV DETERMINATION OF MANGANESE

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Indian Standard

METHODS OF TEST FOR PULP

PART XV DETERMINATION OF MANGANESE

0. FOREWORD

0.1 This Indian Standard (Part XV) was adopted by the Indian Standards Institution on 30 August 1975, after the draft finalized by the Paper and Its Products (Excluding Packaging Materials) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard is mainly based on ISO/R 1830-1970 'Pulps — Determination of manganese', issued by International Organization for Standardization.

0.3 Copper, iron and manganese, if present in pulp in more than traces, not only discolour it but may also affect the dyeing properties. Manganese on bleaching may even impart a reddish tint. It is, therefore, essential to know the quantity of manganese in the pulp.

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part XV) prescribes the method for the determination of manganese in pulp.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960†), freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of test.

3. PRINCIPLE

3.1 The pulp is ashed and dissolved in nitric acid. Manganese is oxidized to permanganate with sodium periodate followed by colorimetric determination by measuring the optical density at 525 nm.

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*Rules for rounding off numerical values (*revised).*

†Specification for water, distilled quality (*revised).*

4. REAGENTS

4.1 Sodium Sulphide Solution (Na_2SO_3) — 50 g/1.

4.2 Nitric Acid — 1.5 M. Prepared by diluting 100 ml of nitric acid (r. d. 1.4) to 1 litre.

4.3 Periodate-Phosphoric Acid Solution — 50 g ??? sodium periodate ($NaIO_4$) and 200 ml ??? 85 percent phosphoric acid (H_3PO_4) per litre.

4.4 Standard Manganese Solution — 0.1 mg ??? manganese per millilitre. Weigh 0.274 9 g of manganese sulphate ($MnSO_4$), ??? at 300°C, into a 1 litre volumetric flask. Dissolve with water and dilute to the mark.

5. APPARATUS

5.1 Dishes — of platinum, porcelain or quartz.

5.2 Spectrophotometer or Filter Colorimeter

5.3 Cells — for use in the spectrophotometer or filter colorimeter.

6, CALIBRATION

6.1 Dilute the standard manganese solution ten times so that 1 ml corresponds to 0.01 mg of manganese. Pipette aliquots of 1, 2, 5, and 10 ml of the diluted solution into 25 ml volumetric flask. Without any further dilution heat the solutions by placing the flasks in a steam-bath and add 1 ml of the periodate-phosphoric acid solution to each flask. Keep the flasks in the steam-bath for 5 minutes after the addition and then dilute to the mark. Cool to 20 ± 2°C and adjust the volume. The temperatures of the solutions in the series should not differ by more than 3°C. Measure the optical density at 525 ran with a solution containing 1 ml of periodate-phosphoric acid solution and 24 ml of water as a reference. Divide the reading by the length of the cell.

6.2 The manganese concentrations of the coloured solutions are 0.4, 0.8, 2.0 and 4.0 mg of manganese per litre respectively. Plot the optical density values divided by the length of the cell against the manganese concentrations, and check that the points lie on a straight line going through the origin.

7. PREPARATION OF THE SAMPLE

7.1 Tear the air-dry sample into pieces ??? suitable size. Do not use cut of punched edges or other parts where metallic contamination may have occurred.

8. PROCEDURE

8.1 Preparation of Test Sample — Weigh to the nearest 0.01 g about 20 g of pulp (or 10 g for manganese contents above 5 mg/kg) into a dish. At the same time weigh out a separate test sample for the determination of dry matter content.

8.2 Determination — Ash the test sample as described in IS : 6213 (Part VII)-1971*. Add three drops of sodium sulphite solution to the ash ??? dissolve in a maximum ??? 5 ml of nitric acid. Place the dish on a steam-bath and evaporate to dryness.

8.2.1 Add a few drops of nitric acid and transfer the contents of the dish with the aid of water to a 25-ml volumetric flask. Heat the contents by placing the flask in a steam-bath. Add 1 ml of the periodate-phosphoric acid solution and let the flask remain in the steam-bath for 5 minutes after the addition. Dilute with water to the mark, cool to 20 ± 2°C and again adjust the volume to the mark. The temperature should not differ by more than 3°G from that of the manganese solutions used for the calibration. If necessary, centrifuge the solution *(see Note)*. Measure the optical density at 525 nm with a solution containing 1 ml of periodate-phosphoric acid solution and 24 ml of water as a reference. Divide the reading by the length of the cell.

NOTE — If the solution is turbid, the turbidity may be removed by centrifuging.

9, CALCULATION

9.1 Carry out two determinations and calculate the manganese content, expressed as milligrams per kilogram ??? pulp, by the following formula:

$$\text{Manganese content in milligrams per kilogram (parts per million)} = \frac{a \times V}{m}$$

where

a = the amount ??? manganese in milligrams per litre ??? coloured solution, obtained from the optical density values and the calibration curve;

V = volume in ml ??? the coloured solution; and

m = mass in g ??? pulp, calculated on an oven dry basis.

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*Methods of test for pulp: Part VII Ash content in pulp.

Report the result as the mean of the two determinations to the first decimal place.

NOTE — The precision of the method as such is high. Thus, the difference between results of duplicate tests is usually attributable to uneven distribution of the trace metal in the pulp.

10. TEST REPORT

10.1 The test report should give the following information:

a) Precise identification of the sample;
b) The results and the form in which they are expressed;
c) Any particular points observed in the course of the test; and
d) Any details of procedure not specified in this standard, or optional; which might affect the results.

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METHODS OF TEST FOR PULP, PAPER AND ALLIED PRODUCTS

IS:

1060 (Part I)-1966 Methods of sampling and test for paper and allied products: Part I (*revised*)
1060 (Part II)-1960 Methods of sampling and test for paper and allied products: Part II
1060 (Part III)-1969 Methods of sampling and test for paper and allied products: Part III
2188-1962 Methods of test for paper for electrical purposes
4006 (Part I)-1966 Methods of test for paper and pulp based packaging materials, Part I
4006 (Part II)-1972 Methods of test for paper and pulp based packaging materials, Part II
5285-1969 Methods of test for fibre analysis of paper and board
6213 (Part I)-1971 Methods of test for pulp: Part I Water solubility of pulp
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6213 (Part IV)-1971 Methods of test for pulp: Part IV Determination of viscosity of pulp
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6213 (Part VII)-1971 Methods of test for pulp: Part VII Ash content in pulp
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6213 (Part XI)-1975 Methods of test for pulp: Part XI Determination of acid insoluble ash
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